Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP)

Liquids Coatings – Generic Testing and Quality Assurance Protocol

Revision No. 0

February 16, 2000

U.S. EPA - U.S. Army ARDEC/ETO Interagency Agreement No.: DW21938366
U.S. Army TACOM-ARDEC NDCEE Contract No.: DAAE30-98-C-1050
Task No.: 208
SOW Task No.: 3

Prepared by
National Defense Center for Environmental Excellence (NDCEE)

Operated by Concurrent Technologies Corporation

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Submitted by

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SI to English Conversions

SI Unit	English Unit	Multiply SI by factor to obtain English
°C	°F	(1.80 E + 00), then add 32
L	gal. (U.S.)	2.642 E - 01
m	ft	3.281 E + 00
kg	lbm	2.205 E + 00
kPa	psi	1.4504 E - 01
cm	in.	3.937 E - 01
mm	mil (1 mil = 1/1000 in.)	3.937 E + 01
m/s	ft/min	1.969 E + 02
kg/L	lbm/gal. (U.S.)	8.345 E + 00

List of Abbreviations and Acronyms

ACS	American Chemical Society
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
CBD	Commerce Business Daily
CCEP	Coatings and Coating Equipment Program
CTC	Concurrent Technologies Corporation
DFT	dry film thickness
DI	deionized
DOI	distinctness-of-image
EPA	U.S. Environmental Protection Agency
ETF	Environmental Technology Facility
ETV	Environmental Technology Verification Program
GC/MS	gas chromatography/mass spectrometry
HAP	hazardous air pollutant
IR	infrared
MEK	methyl ethyl ketone
MSDS	Material Safety Data Sheet
NDCEE	National Defense Center for Environmental Excellence
NIST	National Institute for Standards and Technology
PLC	programmable logic controller
QA/QC	quality assurance/quality control
QMP	Quality Management Plan
RFT	request for technologies
SOP	standard operating procedure
TQAPP	Testing and Quality Assurance Project Plan
UV	ultraviolet
VOC	volatile organic compound
WBS	work breakdown structure

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1.0 INTRODUCTION

1.1 Purpose of the Generic Testing and Quality Assurance Protocol

The primary purpose of this document is to establish the generic protocol for liquid coatings. The secondary purpose is to establish the generic format and guidelines for Liquid Coatings Testing and Quality Assurance Project Plans (TQAPPs).

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) project level TQAPPs will establish the specific data quality requirements for all technical parties involved in each project. A defined format, as described below, is to be used for all ETV CCEP TQAPPs to facilitate independent reviews of Project Plans and test results, and to provide a standard platform of understanding for stakeholders and participants.

1.2 Quality Assurance Category for the ETV CCEP

Projects conducted under the auspices of the ETV CCEP will meet or exceed the requirements of the American National Standards Institute/American Society for Quality Control (ANSI/ASQC), Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, ANSI/ASQC E-4 (1994) standard. This protocol will ensure that project results are compatible with and complementary to similar projects. ETV CCEP Liquid Coating TQAPPs will be adapted from this standard and the ETV Program Quality Management Plan (QMP). These TQAPPs will contain sufficient detail to ensure that measurements are appropriate for achieving project objectives, data quality is known, and that the data are legally defensible and reproducible.

1.3 Logic and Organization of the Protocol Document

This liquid coating protocol document contains the sections outlined in the ANSI/ASQC E-4 standard. As such, this protocol identifies processes to be used, test and quality objectives, measurements to be made, data quality requirements and indicators, and procedures for recording, reviewing, and reporting data.

The major technical sections to be discussed in this protocol are as follows:

- Project Description
- Project Organization and Responsibilities
- Quality Assurance (QA) Objectives
- Site Selection and Sampling Procedures
- Analytical Procedures and Calibration
- Data Reduction, Validation, and Reporting
- Internal Quality Control (QC) Checks
- Performance and System Audits
- Calculation of Data Quality Indicators
- Corrective Action
- Quality Control Reports to Management
- References
- Appendices.

1.4 Formatting

In addition to the technical content, this protocol also contains standard formatting elements required by the ANSI/ASQC E-4 standard and Concurrent Technologies Corporation (*CTC*) deliverables. Standard format elements include, at a minimum, the following:

- Title Page
- TQAPP Approval Form
- Distribution List
- Table of Contents (with an explanation of any deviations from Category II required elements)
- Document Control Identification (in the plan header)

  Section No. ________
  Revision No. ________
  Date: ________
  Page: __ of __

1.5 Approval Form

Key ETV CCEP personnel will indicate their agreement and common understanding of the project objectives and requirements by signing the TQAPP Approval Form for each liquid coating tested. Acknowledgment by each key person indicates commitment toward implementation of the plan. Figure 1 shows the Approval Form format to be used.

APPROVAL FORM

Date Submitted: ______________ QTRAK No.: ____________________

Revision No.: ______________ Project Category: ____________________

Title: ____
____
____
____

Project/Task Officer: ____

EPA/Address/Phone No.: ____

U.S. EPA - U.S. Army ARDEC/ETO Interagency Agreement No.: __________	U.S. Army TACOM- ARDEC NDCEE Contract No.: __________	Task No.: _____

APPROVALS

CTC Project Manager	Signature	Date
CTC QA Officer	Signature	Date
ETV EPA Pilot Manager	Signature	Date
ETV EPA Pilot QA Manager	Signature	Date

CTC - Concurrent Technologies Corporation

Figure 1. Testing and Quality Assurance Project Plan Approval Form

2.0 PROJECT DESCRIPTION

2.1 General Overview

The ETV CCEP was established as an unbiased, third-party verification organization that could provide pertinent test data of environmentally friendly coatings and coating equipment so that they may penetrate the industry faster, thereby facilitating environmental improvements. Organic finishing processes are used by many industries to impart protection and aesthetic appeal to their products. Organic coatings contribute nearly 20 percent of total stationary area source volatile organic compound (VOC) emissions, as well as a significant percentage of toxic air emissions such as hazardous air pollutants (HAPs). Coatings are continually being developed or modified to reduce their impact on the environment. This is primarily accomplished by lowering VOC/HAP content in the paint formulation and using chemicals that have a reduced tendency to form and release VOCs/HAPs to the environment during application and curing. Often, these coatings are slow to penetrate the market because potential users, especially small companies, do not have the resources to test the new coatings for their particular application and may be skeptical of the coating vendors' claims.

The ETV CCEP, a joint venture of the U.S. Environmental Protection Agency (EPA) and Concurrent Technologies Corporation (*CTC*) of Johnstown, Pennsylvania, in conjunction with the National Defense Center for Environmental Excellence (NDCEE) Program, has been established to provide unbiased, third-party data. The ETV CCEP has been tasked to develop, and subsequently utilize, a series of standardized protocols to verify the performance characteristics of coatings and coating equipment. The objectives of this particular protocol are to 1) quantify the VOC and HAP content of the liquid coatings and 2) verify the quality and durability performance of the liquid coatings. This protocol will verify the performance of liquid coatings applied substrates such as: metals, plastics, wood, or composites.

To maximize the ETV CCEP's exposure to the coatings industry, the data from the verification testing will be made available on the Internet at the EPA's ETV Program website (*http://www.epa.gov/etv/*) under the P2 Innovative Coatings and Coating Equipment Pilot, as well as through other sources (e.g., publications and seminars). This will help establish the ETV CCEP's reputation in the private sector. A long-range goal of this initiative is to become a vital resource to the industry and self-sustaining through private support. This is in addition to its primary objective of improving the environment by rapidly introducing more environmentally friendly coatings into industry.

2.1.1 Demonstration Factory Testing Site

CTC has been tasked under the NDCEE Program to establish a demonstration factory capable of prototyping processes that will reduce or eliminate environmentally harmful materials used or produced in manufacturing. To accelerate the transition of environmentally friendly processes to the manufacturing base, *CTC* offers the ability to test processes and products on full-scale, commercial equipment. This demonstration factory is a major national asset. It includes a combination of organic finishing, cleaning, stripping, inorganic finishing, and recycle/recovery equipment. The organic finishing equipment in the demonstration factory will be available for the pilot-scale testing performed in this project, (e.g., surface pretreatment, powder coating, electrocoating, wet spray, and conventional and infrared cure ovens). Ancillary equipment for plating, nonhalogenated cleaning, and nonchromate conversion coating are also available. Layouts of the *CTC* Demonstration Factory and the organic finishing line are shown in Figures 2 and 3, respectively.

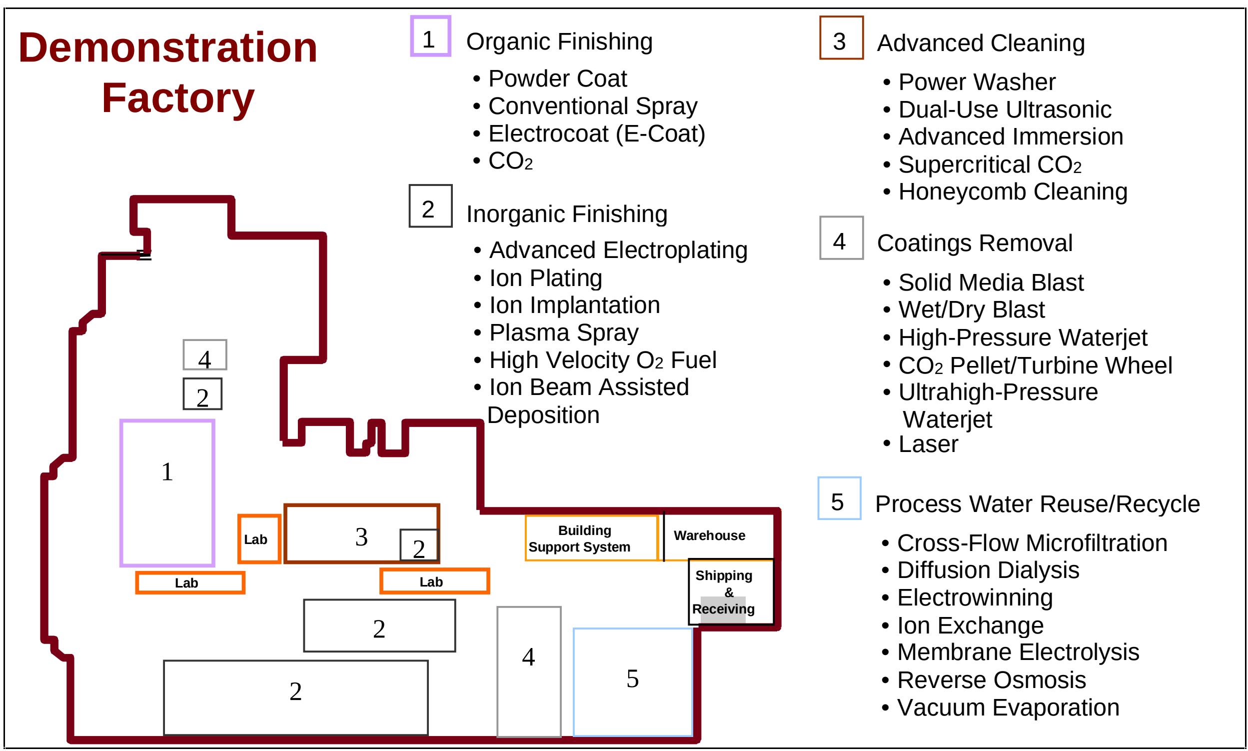


Figure 2. *CTC* Demonstration Factory Layout

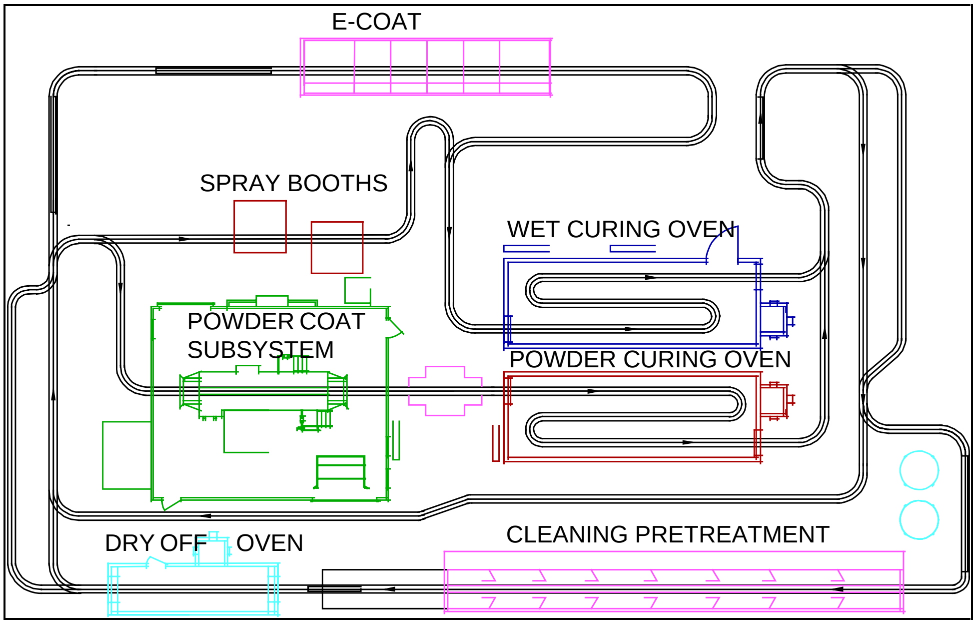


Figure 3. Demonstration Factory Organic Finishing Line

Depending on the testing required, other facilities and laboratories may be used for capabilities not available at *CTC*. In that case, the ETV CCEP will ensure that the requirements of the TQAPP and all associated QA procedures are completed.

2.1.2 Laboratory Facilities

In support of the demonstration factory organic finishing processes, *CTC* maintains extensive state-of-the-art laboratory testing facilities. These laboratory facilities are used for the measurement and characterization of processes and specimens, as well as bench-scale coating technology evaluations. Table 1 lists the various testing and evaluation laboratories, as well as representative equipment holdings that are relevant to ETV CCEP verification projects.

Table 1. Testing Laboratories and Representative Laboratory Equipment Holdings

Laboratory	Focus	Laboratory Equipment
Environmental Testing	1) Identification and quantification of biological, organic, and inorganic chemicals and pollutants to all media. 2) Industrial process control chemical analysis	Hewlett Packard 5972A GC/MS Varian Liberty 110 Sequential ICP P-E 4100ZL Graphite Furnace Mitsubishi GT06 Autotitrator P-E Headspace GC/ECD/FID TOC/Flashpoint/pH/Conductivity Graseby 2010 Isokinetic Stack Analyzer Graseby 2800 VOST Stack Sampler Questron Q-Wave 1000 Microwave Leeman PS200/AP200 Mercury Stations Millipore TCLP/ZHE Extraction Station Lachat Quickchem Flow Injection Analyzer
Destructive and Nondestructive Evaluation	Evaluation of product and process performance, and surface cleanliness	Optically Stimulated Electron Emission X-ray/Magnetic/Eddy Current Thickness Salt Spray Corrosion Chamber Microhardness/Tensile/Fatigue/Wear
Materials and Mechanical Testing	Measurement of service and processing material and mechanical properties	Noran and CAMScan Electron Microscopes Leco 2001 Image Analysis System Nikon and Polaroid Light Optical Microscopes EDAX Energy Dispersive Spectrometer Single Crystal Imaging Metallography Polishing/Grinding/Etching MTS Machines Tinius Olsen Testers Impact Testers
Powder Metallurgy	Investigation of powder properties	Horiba LA900 Laser Particle Size Analyzer Autopore II 9020 Mercury Porosimeter Accupyc 1330 Pycnometer Gemini II 2370 Surface Area Analyzer
Intelligent Processing of Materials	Development and evaluation of embedded process sensors	TEC Model 1600 Stress Analyzer Spectraphysics Argon & ND:YAg Lasers Resonance Frequency System
Risk and Environment Analysis	Management, monitoring, and evaluation of material and process alternatives from health and safety perspective	Biosym: molecular modeling software MOPAC, Extend, HSC Chemistry, Riskpro, Sessoil, GIS
Calibration Laboratory	Calibration of equipment, sensors, and components to nationally traceable standards	Transmation Signal Calibrator (milliamps, millivolts) Thermacal Dry Block Calibrator (Temperature) Druck Pressure Calibrator (Pressure) Fluke Digital Multimeter (Voltage)

2.1.3 Off-Site Testing

While *CTC* 's Demonstration Factory is adequately equipped with a variety of organic finishing equipment, it may not possess certain liquid coating application equipment that is specified by a liquid coatings vendor; therefore, if a liquid coating vendor specifies the use of application equipment not possessed by *CTC*, *CTC* may choose to procure the required equipment or conduct verification testing at an offsite facility. If testing is conducted offsite, the verification testing will be controlled and observed by *CTC* technical personnel. The facility will be chosen by *CTC*, and it must meet the standards of the individual TQAPP, ETV CCEP Quality Management Plan (QMP), and ETV Program QMP.

Each TQAPP will specify how the test parts are pretreated and cleaned, coated (liquid coating), cured, and analyzed for response factors. If the offsite facility is unable to pretreat the parts, pretreated parts will be purchased or *CTC* will pretreat the parts in its facility. In both scenarios, the parts will be shipped to the testing site by *CTC*. Otherwise, the offsite facility will clean, coat, and cure the parts and then ship them to *CTC*. *CTC* will perform the laboratory analyses of the critical response factors (see section 2.2.9). The offsite testing personnel will document all critical and noncritical control factors and qualitative, noncritical control factors (see section 2.2.9) at the offsite testing facility.

2.1.4 Statement of Project Objectives

The overall objective of the ETV CCEP is to verify pollution prevention characteristics and/or performance of coatings and coating equipment technologies, and to make the results of the verification tests available to prospective technology users. The ETV CCEP aspires to increase the use of more environmentally friendly technologies in products finishing in an effort to reduce emissions. Analysis methods used for these tests will follow those developed by the American Society for Testing and Materials (ASTM) or similarly accepted method.

The primary criteria for verification of liquid coatings will be:

- Does the coating provide a significant environmental benefit in terms of reduced VOC/HAP content and/or reduced emissions during application?
- Pending method approval, does the coating provide a significant environmental benefit in terms of reduced VOC/HAP formation/emissions during curing?
- Does the coating provide an acceptable finish?

Based on the best available data, as presented by an unbiased third party, end-users will be able to determine whether the coating can provide them with a pollution prevention benefit while meeting the finish quality requirements of their application. This program intends to supply end-users with the unbiased technical data to assist them in this decision-making process.

The pollution prevention potential of liquid coatings is the primary reason the coatings are being verified by this program. Liquid coatings vendors have been invited to meetings at the U.S. EPA facilities in Research Triangle Park to discuss the potential benefits of the program.

The quantitative pollution prevention benefit in terms of reducing or eliminating VOC/HAP emissions depends on a multitude of factors; therefore, the liquid coating will be applied per the coating vendor's instructions, and the resulting verification data will be representative of the exact conditions tested. To qualify the existence of an environmental benefit, this program will conduct a test to quantify the VOC/HAP content from liquid coatings as a surrogate for measuring VOC/HAP emissions. The resulting data will be presented and compared to data from existing coatings used in the liquid coating's target industry, as appropriate. The ETV CCEP will provide the funding for assessing the finish quality and environmental impact of those existing coatings.

2.1.5 Technical/Experimental Approach and Guidelines

A schematic diagram of the verification process is shown in Figure 4, and the tasks planned for this project are listed below:

- Conduct initial stakeholders meeting.
- Investigate/identify/prioritize focus areas.
- Identify technology vendors.
- Develop technology solicitation – Commerce Business Daily (CBD) Announcement/Request for Technologies (RFT).
- Issue technology solicitation.
- Review responses to solicitation.
- Review generic protocol by stakeholders and technology vendors.
- Develop and obtain EPA approval of the generic protocol document.
- Obtain program participation commitment from vendors.
- Provide TQAPPs for each liquid coating tested.
- Obtain liquid coating vendor's concurrence with the TQAPP.
- Obtain *CTC* and EPA approval of TQAPPs.
- Conduct coating baseline test, as appropriate.
- Conduct verification test of each liquid coating.
- Prepare and provide draft test reports to EPA.
- Prepare and provide final test reports to EPA.
- Verification Statements – issued by *CTC*.

Each TQAPP is dependent upon the specific liquid coating being tested. Nevertheless, some general guidelines and procedures can be applied to each TQAPP. Table 2 describes these general guidelines and procedures.

Table 2. Overall Guidelines and Procedures to be Applied to the TQAPP

• A detailed description of each part of the test will be given. This will include a detailed Design of Experiments, and a schematic diagram of testing to be performed. • Critical and noncritical factors will be listed. Noncritical factors will remain constant throughout the testing. Critical factors will be listed as control (process) factors or response (coating product quality) factors (see Section 2.2.9). • The TQAPP will identify the testing site. • Regardless of where the testing is performed, the ETV CCEP will ensure that the integrity of third-party testing is maintained. • Regardless of where the testing is performed, the QA portion of the Generic Protocol will be strictly adhered to. • A statistically significant number of samples will be analyzed for each critical response factor (see Table 5). Variances (or standard deviations) of each critical response factor will be reported for all results.

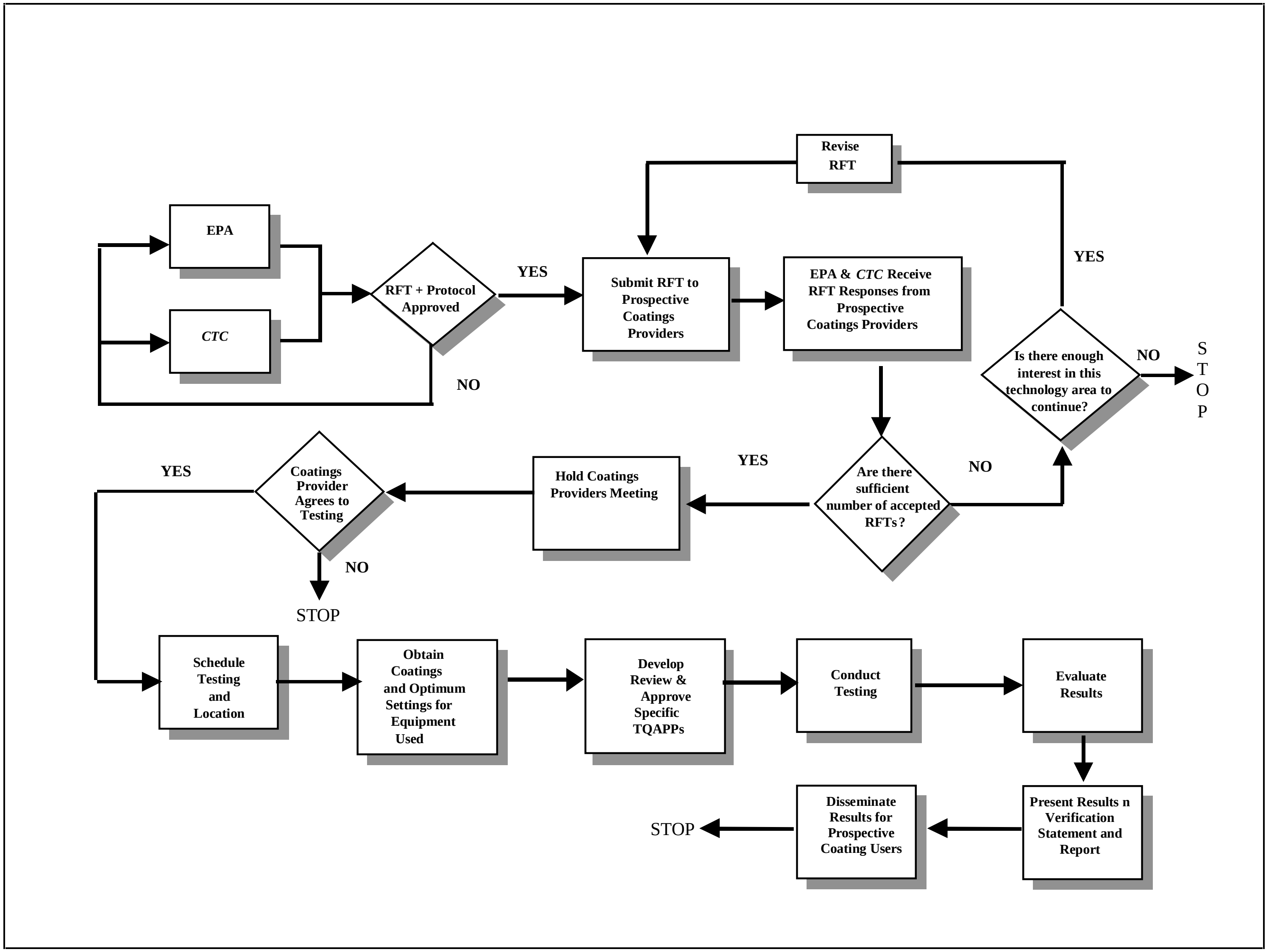


Figure 4. Schematic of the Verification Process

2.2 Verification Test Description

2.2.1 Test Approach

The following approach will be used in the test protocol:

- Performance parameters to be verified will be determined.
- A standard test panel (and possibly other products) will be chosen that will enable thorough testing of coating performance.
- Coating manufacturers will supply the test coating and the optimum equipment settings for application and curing.
- A verification test will be completed to determine the performance of the liquid coatings.
- A statistically valid test program that efficiently accomplishes the required objectives will then be used to analyze the test results.

2.2.2 Verification Test Objectives

The objectives of the verification tests performed per this protocol are to determine the VOC and HAP content and to verify the quality and durability of liquid coatings. In addition, the VOC/HAP emissions generated during the curing of the coating may be checked upon EPA approval of a method for determining those emissions. The coated test panels will be checked for dry film thickness (DFT), visual appearance, and at least three of the following analyses: gloss, color, distinctness-of-image (DOI), adhesion, corrosion resistance, impact resistance, flexibility, hardness, humidity resistance, weather resistance, wear resistance, and resistance to methyl ethyl ketone (MEK). The cost associated with each analysis (except the mandatory DFT and visual appearance) will be presented to the participating vendors. The coating vendors will then choose which optional tests they want to have performed on the panels prepared using their coating. The coating vendor must choose a minimum of three optional tests. The total cost for completing each verification test and the vendor's share of that cost will depend on the number and type of analyses chosen. Additional pretreatment processes or tests that are either listed above or requested by the vendor may be included at the expense of the liquid coating vendor.

2.2.3 Standard Test Panel

The actual test panels may be fabricated from steel, stainless steel, glass, plastic, alloys, wood, or composites based on the liquid coating vendor's recommendations. However, the default standard test panel, as is shown in Appendix C, *Standard Test Panel*, will be 30.5 cm (12 in.) long and 10.2 cm (4 in.) wide with 0.6-cm (0.25-in.) hole punched in one end so that it may be suspended from a hook. Other parts may be treated and tested at the expense of the liquid coating vendor. (***Note:*** *CTC*'s Organic Finishing Line can accommodate parts with dimensions up to 1.2 m x 1.2 m x 0.9 m (4 ft x 4 ft x 3 ft), and parts weighing up to 113.5 kg (250 lbs).)

2.2.4 Coating Specification

The liquid coatings submitted for verification testing should provide an environmental benefit over the existing coatings currently in use in each liquid coating's target industry. The liquid coatings will also be reviewed by the stakeholders group to determine their status as innovative coatings.

Each coating vendor will supply its test coating and respective specifications for the verification test. In addition, each vendor will supply a sufficient amount of coating to complete the verification tests, the exact preparation instructions, and the instructions/parameters for applying the coating. The application procedures and conditions must be typical of the real target industry.

2.2.5 Standard Coating Application Apparatus

This protocol outlines the default application apparatus to be used for liquid coating verification tests. The default application method atomizes the liquid coating via automated spray application equipment. Off-site automated equipment and equipment setup will be specified in each individual TQAPP, if applicable. The information contained in this protocol describes a standard apparatus setup for verification tests conducted at *CTC*. Appendix A shows the selected apparatus setup, and Appendix B, shows the testing location relative to the Organic Finishing Line. Unless otherwise indicated by the vendor, testing will be performed in one of the liquid spray booths in *CTC*'s Demonstration Factory.

Prior to testing, the coating vendors will be supplied with important information such as the average ambient temperature, ambient humidity, and substrate temperature. The coating vendors will then determine the operating parameters of the spray equipment (e.g., input air pressure, gun-to-target distance, horizontal gun speed, flash time, dwell time).

Panel pretreatment is specific to the substrate material and will be specified by the liquid coatings vendor. If panels are not purchased in a pretreated condition, pretreatment will be performed at *CTC*. The pretreatment sequence typically conducted at *CTC* is as follows:

1. Panels receive an alkaline cleaning followed by a fresh water rinse.
2. Zinc phosphate or other recommended treatment is applied followed by a water rinse.
3. Nonchromate or other recommended sealer is applied followed by a DI water rinse.
4. Panels are dried in a dryoff oven.

One random test panel will be removed for pretreatment analysis for each verification test; therefore, eight additional panels will be pretreated for each verification test to be used for the pretreatment analysis and as setup panels for the test. Any pretreatment conducted by an offsite facility is expected to follow a similar sequence.

Standard test panels will be suspended from racks containing a single row of up to eight panels per rack, as shown in Appendix A, *Apparatus Setup*. The test panels will be fixtured on the rack to minimize movement during spraying. Fixturing consists of a flat bar that connects all eight hooks. The bar will minimize side-to-side rotation of the panels. A second bar is oriented near the bottom of the panels to prevent the bottom of the panels from moving away from the gun. The test panels will be transported to the spray booth by an overhead conveyor. A mechanical stop mechanism will align the racks of test panels in the proper position relative to the spraying mechanism. Once the racks are in position, the programmable logic controller (PLC) of the spraying mechanism will activate the motors that drive the linear motion translators. The translators will move both horizontally and vertically, enabling the application equipment to treat an area approximately 1.4 m by 1.4 m (4.5 ft. x 4.5 ft.). The panels will be automatically sprayed using vertical overlap of the spray pattern. The PLC will also trigger the pneumatic spray gun or a pneumatically actuated cylinder that compresses the trigger of a manual spray gun. During dwell time between passes, paint flow will be interrupted to minimize paint usage. Once the spray application is complete, the PLC will release the mechanical stop that maintains the position of the rack, enabling the overhead conveyor to move the next rack into position.

Before each test, a set of dummy panels will be coated to ensure that the equipment parameters are set correctly. (The coating vendor may wish to assist in this step.) The input air pressure will be monitored throughout the test, and the air pressure at the cap and air horns (if applicable) will be measured using a verified test cap prior to each run. The paint usage will be determined through gravimetric means or by the use of an in-line flow meter, as appropriate.

To help ensure proper equipment setup and operation, the liquid coatings vendors will be invited to participate in the startup phase of the testing and to observe the testing of their coatings.

The pressure drop across the filters will be checked prior to each run and at the end of the test. The pressure drop is monitored in the event that the filter bank system malfunctions. A pressure drop across the filter bank greater than 1 cm (0.4 in.) of water shall indicate that the system requires service and/or changing of the filters.

2.2.6 Coating Baseline Test

A coating baseline test may be performed for a coating that submitted for verification, as appropriate. The coating baseline will be used to determine the relative environmental and performance benefits of the liquid coating being verified. The coating baseline panels will also be evaluated for DFT, visual appearance, and the same optional tests chosen by the coating vendor for the verification test.

The coating baseline will use an existing coating and application method that is consistent with the verified liquid coating's target industry. The coating baseline testing will be designed and performed by the ETV CCEP personnel. Certain operating parameters used for the coating baseline will be identical to the parameters used for the liquid coating verification test. Other parameters will be developed from the application equipment's or coating manufacturer's recommendations and/or experimental trials performed by the ETV CCEP.

2.2.7 Design of Experiment

This test protocol will verify the performance of liquid coatings submitted in response to the associated CBD or RFT. A mean value and variance (or standard deviation) will be reported for each critical response factor. If a liquid coating vendor makes a claim about a particular coating characteristic, the owner of the coating will be asked to submit a confidence limit and specification limit (acceptable quality limit) for that claim for verification purposes. If the owner does not submit a confidence and specification limit, a default 95% confidence limit will be applied.

Any claims made by the coating vendor regarding particular coating characteristics will be used in the design of experiments. The appropriate number of test panels to be coated and analyzed will be based on the confidence limit, specification limit, and the appropriate statistical test to be applied to the results (i.e., Student's T-Test, Chi Square Test, or F-Test). Each verification test will consist of five runs with one rack of eight panels in a single row per run. The statistical analyses for all response factors will be carried out using Minitab statistical software.

Prior to the verification test, setup panels will be coated to ensure that the equipment parameters are correct. In actual verification testing, one panel will be used for pretreatment analysis, and 40 panels will be coated to determine the pollution prevention benefit and finish quality. Specifically, the standard test panels coated during the verification test will be analyzed for their chemical and physical properties as well as appearance.

If requested in the RFT or CBD response, the coating vendor can supply five additional parts to be coated during each verification test run. Fixturing of parts will be determined after parts are submitted by the coating supplier, and suppliers are bound to the part size and weight restrictions identified in Section 2.2.3.

2.2.8 Performance Testing

Liquid coating vendors will provide the ETV CCEP with coating specifications and appropriate equipment settings. The ETV CCEP will not attempt to optimize test settings during the actual test runs; however, the coating vendors will be given the opportunity to do so during the startup phase of the testing. The ETV CCEP will provide the liquid coating vendors with a list of key noncritical test factors that may affect the critical response factors (i.e., test results).

All testing will be conducted on the coated standard panels. All such tests will be performed per ASTM procedures and provide insight to the chemical and physical properties of the coatings. A comparison will be made from panel to panel, rack to rack, and run to run.

2.2.9 Critical and Noncritical Factors

In a designed experiment, critical and noncritical control factors must be identified. In this context, the term "critical" does not convey the importance of a particular factor. (Importance can only be determined through experimentation and characterization of the total process.) Rather, this term displays its relationship within the design of experiments. For the purposes of this protocol, the following definitions will be used for critical control factors, noncritical control factors, and critical response factors.

- Critical control factor – a factor that is varied in a controlled manner within a design of experiments matrix to determine its effect on a particular outcome of a system.
- Noncritical factors – factors that remain relatively constant or are randomized throughout the testing.
- Critical response factors – the measured outcomes of each combination of critical and noncritical control factors used in the design of experiments.

In the case of the verification testing of a coating, there is only one critical control factor, and that is the coating itself. All other processing factors are noncritical control factors; therefore, the multiple runs and sample measurements within each run for each critical response factor will be used to determine the amount of variation expected for each critical response factor. For example, for each coating application, parameters associated with pretreatment would remain constant, and, thus, be noncritical control factors; however, a parameter, such as adhesion, would be identified as a critical response factor and could vary from run to run.

Tables 3 through 5 identify the factors to be monitored during testing, as well as their acceptance criteria (where appropriate), data quality indicators, measurement locations, and measurement frequencies. The values in the Total Numbers column are based on the default test scenarios.

Table 3. Critical Control Factors

Critical Control Factor	Resin Type	Solvent Type	Cure Method	Target Industry
Liquid Coating	TBD	TBD	TBD	TBD

TBD - To Be Determined

Table 4. Noncritical Control Factors

Noncritical Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number for Each Test
Application Method Manufacturer/Model	from coating provider	Factory floor	Continuous	N/A
Input Air Pressure Gun/Pot	from coating provider	Factory floor	Continuous	N/A
Products involved in Testing	Standard test panels (material TBD)	N/A	See default scenario for each test in Section 5.3	40 panels
Pretreatment Weight	TBD g/m^2	Random uncoated panel	1 Standard test panel per test	1
Surface Area of Each Panel Coated	TBD (cm^2 or $in.^2$)	Top and right edge of panel	1 Standard test panel	1
Ambient Factory Relative Humidity	< 60% RH	In the factory	Once each run	5
Ambient Factory Temperature	21.1 – 26.7°C	In the factory	Once each run	5
Spray Booth Relative Humidity	< 60% RH	Inside the spray booth	Once each run	5
Spray Booth Temperature	21.1 – 26.7°C	Inside the spray booth	Once each run	5
Spray Booth Air Velocity	0.2-0.5 m/s (40–100 ft/min)	Inside the spray booth	Once per test	1
Distance to Panels	TBD (cm or in.)	Factory floor	Once per test	1
Temperature of Panels, as Coated	21.1 – 26.7°C	Factory floor	Once per run	5
Horizontal Gun Traverse Speed	from coating vendor	Factory floor	Once per test	1
Vertical Drop Between Passes	from coating vendor	Factory floor	Once per test	1
Dwell Time Between Passes	from coating vendor	Factory floor	Once per test	1
Density of Applied Coating	from coating vendor	Sample from coating pot	1 sample each run	5
% Solids of Applied Coating	from coating vendor	Sample from coating pot	1 sample each run	5
Coating Temperature, as Applied	from coating vendor	Sample from coating pot	1 sample each run	5
Coating Viscosity, as Applied	from coating vendor	Sample from coating pot	Before and after run	10
Paint Flow Rate	from coating vendor	Factory floor	Once each run	5
Total Paint Flow	from coating vendor	Factory floor	Once each run	5
Oven Temperature	from coating vendor	Factory floor	Continuous	N/A
Oven Cure Time	from coating vendor	Factory floor	Once each run	5

TBD - To Be Determined

Table 5. Critical Response Factors

Critical Response Factor	Measurement Location	Frequency	Total Number
Environmental			
Volatile Organic Compound Content of the Liquid Coating	see Section 2.2.10	5 samples from liquid coating lot to be used during test	5
Hazardous Air Pollutant Content of the Liquid Coating	see Section 2.2.10	5 samples from liquid coating lot to be used during test	5
Quality/Durability (Mandatory)			
Dry Film Thickness (DFT)	From ASTM B 499 (magnetic)	9 points on 1 standard test panel per run	45
Visual Appearance	Entire test panel and entire rack	1 standard test panel per run and 1 per test	6
Quality/Durability (Optional)			
Gloss	from ASTM D 523	3 points on 1 standard test panel per run	15
Color[a]	from ASTM D 1729	1 randomly selected panel per run, 1 test per panel	5
Color[a]	from ASTM D 2244	1 randomly selected panel per run, 1 test per panel	5
Distinctness of Image (DOI)[b]	from ASTM D 5767 Test Method B	1 randomly selected panel per run, 3 tests per panel	15
Adhesion[c]	from ASTM D 3359	1 randomly selected panel per run, 1 test per panel	5
Pencil Hardness[c]	from ASTM D 3363	1 randomly selected panel per run, 1 test per panel	5
Salt Spray	from ASTM B 117	1 randomly selected panel per run, 1 test per panel	5
Impact	from ASTM D 2794	1 randomly selected panel per run, 1 test per panel	5
Flexibility (Mandrel Bend)	from ASTM D 522	1 randomly selected panel per run, 1 test per panel	5
MEK Rub	from ASTM D 5402	1 randomly selected panel per run, 1 test per panel	5
Humidity Resistance	From ASTM D 1735	1 randomly selected panel per run, 1 test per panel	5
Weather Resistance	From ASTM G 26	1 randomly selected panel per run, 1 test per panel	5
Abrasion Resistance	From ASTM D 4060	1 randomly selected panel per run, 1 test per panel	5

[a] Both color analyses will use the same panel if both are selected.
[b] Except that the sliding combed shutter is replaced by a rotating eight-bladed disc.
[c] The adhesion and pencil hardness tests will all be performed on the same panel as the DFT test.

Qualitative, noncritical control factors used in the verification tests include:

• Equipment preparation	from coating vendor
• Flash time between coats	from coating vendor
• Number of passes	from coating vendor
• Spray pattern	from coating vendor
• Target DFT	from coating vendor.

2.2.10 Determination of VOCs and HAPs

The VOC and HAP content of the liquid coatings will be determined by EPA Methods 24 and 311. To assist in VOC/HAP determinations and assessments, the vendor will be required to submit material safety data sheets (MSDSs) and coating composition information, including the following:

- Total volatile matter
- Coating density
- Solids content
- Water content (as appropriate)
- EPA-exempt solvents content
- Total VOC content
- Specific VOC/HAP identification
- Density of cured coating.

Bulk formulation tests will be performed to assess the quantity of VOCs/HAPs contained in the paint per EPA Method 24. EPA Method 24 requires three separate measurements to determine the total VOC content in the coating. First, the total volatile matter contained within the liquid coating will be determined via gravimetric analysis (ASTM D2369). Next, a Karl Fischer Titration (ASTM D4017 or E1064) will determine water content of the coating, and gas chromatography (ASTM D4457) will determine the amount of exempt solvents. The total VOC content is then calculated, per EPA Method 24, by subtracting the mass of water and mass of exempt solvents from the total volatile matter. Identification of the specific VOCs/HAPs contained in the paint will be performed using gas chromatography/mass spectrometry (GC/MS) per EPA Method 311 or EPA Method 8260B. These analyses may be conducted at an off-site laboratory.

VOC/HAP emissions during curing are a potential environmental concern. However, no industry-accepted standard has been identified to measure such emissions. The U.S. EPA is developing a test method that has the potential to meet the objectives of the ETV Program and industry stakeholders. Until this new method is approved, the ETV CCEP will not analyze the emissions given off during the curing of the liquid coatings. In the event that this new method is approved prior to any verification test, the ETV CCEP reserves the right to incorporate the analysis of cure emissions as part of the critical response factors.

2.3 Schedule

CTC uses standard tools for project scheduling. Project schedules are prepared in Microsoft Project or Primavera, which are accepted industry standards for scheduling. Project schedules show the complete work breakdown structure (WBS) of the project, including technical work, meetings, and deliverables. Each TQAPP will contain an estimated schedule for the verification testing activities.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

CTC employs a matrix organization, with program and line management, to perform projects. The laboratory supports the *CTC* Project Manager and the *CTC* Project Leaders by providing test data. Laboratory Analysts report to the *CTC* Laboratory Manager. The *CTC* Laboratory Manager coordinates with the *CTC* Project Leaders on testing schedules. The *CTC* Project Leaders are the conduit between the laboratory and the *CTC* Project Manager. The *CTC* Project Leaders answer directly to the *CTC* Project Manager. For the ETV CCEP, the *CTC* Project Leaders will be responsible for preparing the TQAPPs and the internal demonstration plans for each test.

The *CTC* QA Officer, who is independent of both the laboratory and the program, is responsible for administering *CTC* policies developed by the Quality Committee. These policies provide for, and ensure that quality objectives are met for each project. The policies are applicable to laboratory testing, factory demonstration processing, engineering decisions, and deliverables. The *CTC* QA Officer reports directly to *CTC* senior management and is organizationally independent of the project or program management activities.

The project organization chart, showing lines of responsibility and the specific *CTC* personnel assigned to this project, is presented in Figure 5. A summary of the responsibilities of each *CTC* participant, his/her applicable experience, and his/her anticipated time dedication to the project during testing and reporting is given in Table 6.

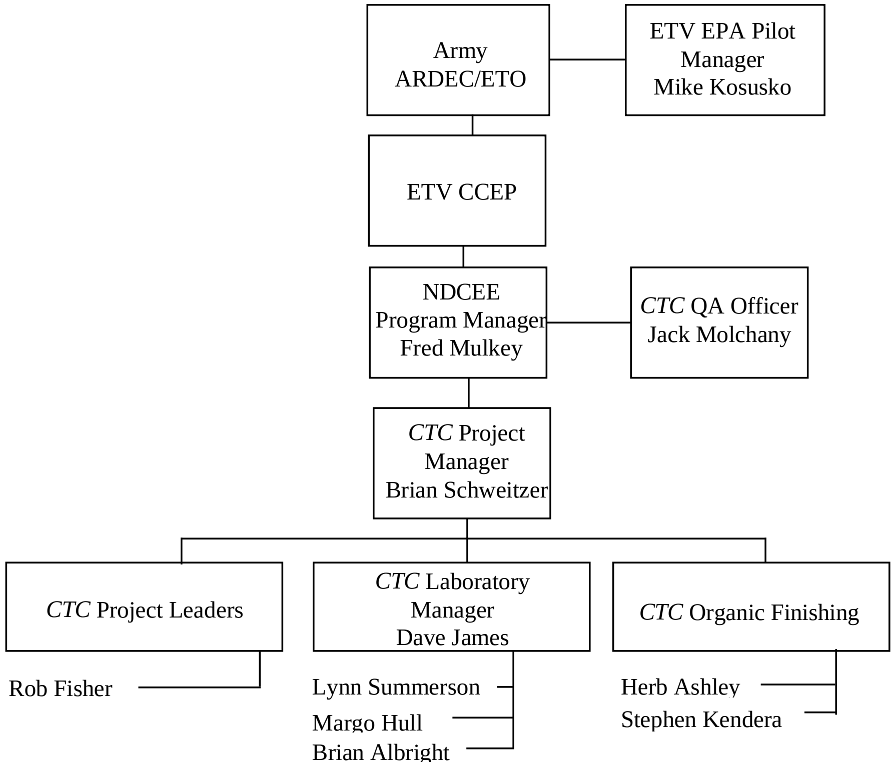


Figure 5. Project Organization Chart

Table 6. Summary of ETV CCEP Experience and Responsibilities

Key *CTC* Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Fred Mulkey – NDCEE Program Manager	Manages NDCEE Program Accountable to *CTC* Technical Services Manager and *CTC* Corporate Management	Laboratory Chemist and Manager (13 years) Project Quality Assurance (13 years) Project Management (12 years) Registered Environmental Manager	M.S., Chemistry B.S., Chemistry	5%
Brian Schweitzer – Manager, Process Engineering/ *CTC* Project Manager	Responsible for overall ETV CCEP technical aspects, budget, and schedule issues on daily basis Accountable to NDCEE Program Manager	Process Engineer (12 years) Project Manager, Organic Finishing (7 years)	B.S., Mechanical Engineering	50%
Jack Molchany – *CTC* QA Officer	Responsible for overall project QA Accountable to NDCEE Program Manager	Industrial QA/QC and (12 years) Quality Mgmt. /ISO 9000 (6 years) Environmental Compliance and ISO 14000 Management Systems (6 years) Certified Hazardous Materials Mgr.	B.S., Industrial Engineering	5%
Rob Fisher – Staff Process Engineer/ *CTC* Project Leader	Technical project support Process design and development Accountable to *CTC* Project Manager	Organic Finishing Regulations (5 years) Organic Finishing Operations (3 years) Professional Engineer	B.S., Chemical Engineering	50%
Herb Ashley – Finishing Engineer	Oversees daily operation of the Organic Finishing Line Provides technical project support. Accountable to *CTC* Project Manager	Organic Finishing Experience (28 years)		10%
Steve Kendera – Sr. Organic Finishing Technician	Performs day-to-day operations of the Organic Finishing Line Accountable to Finishing Engineer	Industrial Paint and Coatings Experience (26 years)		10%
Dave James – Process and Materials Characterization Manager/ *CTC* Laboratory Manager	Coordinates testing laboratory and technical data review Accountable to *CTC* Project Manager, NDCEE Program Manager	Environmental Engineering (17 years) Project/People Management (17 years) ISO 9000/14000 Management Systems (5 years) Certified Industrial Hygienist Certified Hazardous Materials Mgr. Registered Environmental Mgr.	M.S., Environmental Engineering B.S., Ecology	5%
Lynn Summerson – *CTC* Laboratory Leader/Statistical Support Staff	Laboratory analysis Accountable to Lab Manager	Industrial and Environmental Laboratory Testing (18 years)	M.S., Chemistry B.S., Chemistry	20%
Margo Hull – *CTC* Associate Laboratory Leader	Laboratory analysis Accountable to *CTC* Laboratory Manager	Organic/Inorganic Laboratory Testing (6 years)	B.S., Biology	10%
Brian Albright – *CTC* Assistant Laboratory Analyst/ Pretreatment Operator	QC Analysis Accountable to *CTC* Laboratory Manager	Environmental and QC Testing (5 years)	B.S., Chemistry	10%

The *CTC* personnel specified in Figure 5 and Table 6 are responsible for maintaining communication with other responsible parties working on the project. The frequency and mechanisms for communication are shown in Table 7.

Table 7. Frequency and Mechanisms of Communications

Initiator	Recipient	Mechanism	Frequency
NDCEE Program Manager or *CTC* Project Manager	Army ARDEC/ETO and ETV EPA Pilot Manager	Written Report Verbal Status Report	Monthly Weekly
CTC Project Manager	NDCEE Program Manager	Written or Verbal Status Report	Weekly
CTC Laboratory Manager	*CTC* Project Manager	Data Reports	As generated
CTC QA Officer	NDCEE Program Manager	Quality Review Report	As required
ETV EPA Pilot Manager	Army ARDEC/ETO and *CTC*	On-Site Visit	At least once per year

Special Occurrence	Initiator	Recipient	Mechanism/ Frequency
Schedule or Financial Variances	NDCEE Program Manager or *CTC* Project Manager	Army ARDEC/ETO and ETV EPA Pilot Manager	Telephone call, with a written follow-up report as necessary
Major (will prevent accomplishment of verification cycle testing) Quality Objective Deviation	NDCEE Program Manager or *CTC* Project Manager	Army ARDEC/ETO and ETV EPA Pilot Manager	Telephone call with a written follow-up report

4.0 QUALITY ASSURANCE (QA) OBJECTIVES

4.1 General Objectives

The overall objectives of this ETV CCEP protocol is to verify the pollution prevention benefit of liquid coatings and their ability to provide a quality finish. These objectives will be met by controlling and monitoring the critical and noncritical factors, which are the specific QA objectives for this protocol. Tables 3 and 4 list the critical and noncritical control factors, respectively.

The analytical methods that will be used for coating evaluations are adapted from ASTM Standards, or industrial standard equivalent. The QA objectives of the program and the capabilities of these test methods for product and process inspection and evaluation are synonymous because the methods were designed specifically for evaluation of the coating properties under investigation. The methods will be used as published, or as supplied, without major deviations. The specific methods to be used for this project are attached to this document as Appendix D, *ASTM Methods*.

4.2 Quantitative Quality Assurance Objectives

Quality assurance parameters, such as precision, accuracy, and completeness, are presented in Tables 8 and 9. Table 8 presents the manufacturers' stated capabilities of the equipment used to measure noncritical control factors. The precision and accuracy parameters listed are relative to the true value to which the equipment measures. Table 9 presents the precision and accuracy parameters for the measurement equipment for the critical response factors. Precision and accuracy are determined using duplicate analysis and known standards and/or spikes and must fall within the values found in the specific methods expressed.

The Statistical Support Staff, *CTC* QA Officer, and laboratory personnel will coordinate efforts to calculate and interpret the test results.

Table 8. QA Objectives for Precision, Accuracy, and Completeness for All Noncritical Control Factor Performance Analyses

Measurement	Method	Units	Precision	Accuracy[a]	Completeness
Input Air Pressure	Pressure Gauge	psig	±0.2	±5%	90%
Pretreatment Weight	ASTM B 767	g/m^2	±0.005	±0.01	90%
Surface Dimensions of Each Panel Coated	Ruler	cm (in.)	±0.3 (±0.13)	±0.2 (±0.06)	90%
Ambient Factory Relative Humidity	Thermal Hygrometer	RH	±3% of full scale	±3% of full scale	90%
Ambient Factory Temperature	Thermal Hygrometer	°C	±3% of full scale	±3% of full scale	90%
Spray Booth Relative Humidity	Thermal Hygrometer	RH	±3% of full scale	±3% of full scale	90%
Spray Booth Temperature	Thermal Hygrometer	°C	±3% of full scale	±3% of full scale	90%
Spray Booth Air Velocity	per ACGIH[b]	m/s (ft/min)	±0.03[c] (±5)	±0.03[c] (±5)	90%
Distance to Panels	Ruler	cm (in.)	±0.15 (±0.06)	±0.15 (±0.06)	90%
Temperature of Panels, as Coated	IR Thermometer	°C	±0.5% RPD	±1.0%	90%
Horizontal Gun Traverse Speed	Stopwatch	cm/s (in./sec)	±0.001% RPD	±0.001%	90%
Vertical Drop Between Passes	Ruler	cm (in.)	±0.15 (±0.06)	±0.15 (±0.06)	90%
Dwell Time Between Passes	Stopwatch	seconds	±0.001% RPD	±0.001%	90%
Density of Applied Coating	ASTM D 1475	g/l (lb/gal)	±0.6% RPD	±1.8%	90%
% Solids of Applied Coating	ASTM D 2369	%	±1.5% RPD	±4.7%	90%
Coating Temperature, as Applied	Thermometer	°C	±0.5 °C	±0.2 °C	90%
Coating Viscosity, as Applied	ASTM D 1200	seconds	±10% RPD	±10%	90%
Paint Flow Rate	Flow Meter	cm^3/min	±0.5% RPD	±0.5%	90%
Total Paint Flow	Flow Meter	cm^3	±0.5% RPD	±0.5%	90%
Oven Temperature	Thermocoupl e	°C	±2.2 °C	±2.2 °C	90%
Oven Cure Time	Stopwatch	minutes	±0.001% RPD	±0.001%	90%

[a] Accuracy is presented as percent recovery of a standard, unless otherwise noted.
[b] ACGIH – American Conference of Governmental Industrial Hygienists, Inc.
[c] Accuracy and precision stated by the manufacturer for velocities ranging from 20–100 ft/min.
RPD - relative percent difference

Table 9. QA Objectives for Precision, Accuracy, and Completeness for All Critical Response Factor Performance Analyses

Measurement	Method	Units	Precision	Accuracy[a]	Completeness
VOC Content	EPA Method 24	g/l (lb/gal)	±0.6% RPD	±1.8%	90%
HAP Content	EPA Method 311 or 8260B	g/l (lb/gal)	±0.6% RPD	±1.8%	90%
DFT – Magnetic	ASTM B 499	mils[b]	20% RPD	10% True Thickness	90%
Visual Appearance	N/A	N/A	N/A	N/A	100%
Gloss	ASTM D 523	Gloss Units	20% RPD	±0.5	90%
Color--Spectrometer	ASTM D 2244	ΔE Values	20% RPD	± 0.2 ΔE Values	90%
Color--SpectraLight II	ASTM D 1729	Visual	N/A	N/A	90%
DOI[c]	ASTM D 5767 Method B	DOI Units	20% RPD	±3 DOI units	90%
Adhesion	ASTM D 3359 (Tape Test)	Pass/Fail and the 0-5 Rating	All Pass or All Fail	±1 Rating	90%
Pencil Hardness	ASTM D 3363	Pass/Fail	All Pass or All Fail	N/A	90%
Salt Spray	ASTM B117	Pass/Fail	All Pass or All Fail	N/A	90%
Impact	ASTM D 2794 (Direct & Reverse)	Pass/Fail	All Pass or All Fail	Ranges listed in ASTM D 2794	90%
Flexibility	ASTM D 522 (Mandrel Bend)	Pass/Fail	All Pass or All Fail	±15%	90%
MEK Rub	ASTM D 5402	Visual	Being Determined by ASTM	N/A	90%
Humidity Resistance	ASTM D 1735	Pass/Fail	All Pass or All Fail	N/A	90%
Weather Resistance	ASTM G 26	Pass/Fail	All Pass or All Fail	N/A	90%
Abrasion Resistance	ASTM D 4060	Milligrams	46% RPD	Not reported in ASTM D 4060	90%

[a] Accuracy is presented as percent recovery of a standard, unless otherwise noted.
[b] 1 mil = 0.001 inch
[c] Performed by ACT Laboratories, Inc.
N/A = Not Applicable
RPD - relative percent difference

4.2.1 Accuracy

Standard reference materials, traceable to national sources, such as the National Institute for Standards and Technology (NIST) for instrument calibration and periodic calibration verification, will be procured and used where such materials are available and applicable to this project. For reference calibration materials with certified values, acceptable accuracy for calibration verification will be within the specific guidelines provided in the method if verification limits are given; otherwise, 80% to 120% of the true reference values will be used (see Tables 8 and 9). Reference materials will be evaluated using the same methods as for the actual test specimens.

4.2.2 Precision

The experimental approach of this protocol specifies the exact number of test panels to be coated. The analysis of replicate test panels for each coating property at each of the experimental conditions will occur by design. The degree of precision will be assessed based on the agreement of all replicates within a property test group.

4.2.3 Completeness

The laboratory strives for at least 90 percent completeness. Completeness is defined as the number of valid determinations expressed as a percentage of the total number of tests conducted, by test type.

4.2.4 Impact and Statistical Significance Quality Objectives

All laboratory analyses will meet the accuracy, precision, and completeness requirements specified in Tables 8 and 9. The precision will also be checked on test panel replicates to determine whether a nonconformance exists as a result of limitations in the coating technology. If any nonconformance from TQAPP QA objectives occurs, the cause of the deviation will be determined by checking calculations, verifying the testing and measuring equipment, and performing a reanalysis. If an error in analysis is discovered, reanalysis of a new batch for a given trial will be considered, and the impact to overall project objectives will be determined. If the deviation persists despite all corrective action steps, the data will be flagged as not meeting the specific quality criteria, and a written discussion will be generated.

If all analytical conditions are within control limits and instrument and/or measurement system accuracy checks are valid, the nature of any nonconformance may be beyond the control of the laboratory. If, given that laboratory quality control data are within specification and any nonconforming results occur, the results will be interpreted as the inability of the liquid coating undergoing testing to produce parts meeting the performance criteria at the given set of experimental conditions.

4.3 Qualitative QA Objectives: Comparability and Representativeness

4.3.1 Comparability

Liquid coatings will be used at vendor-recommended conditions or conditions otherwise established in agreement with the project stakeholders for each TQAPP. The data will be comparable from the standpoint that other testing programs could reproduce similar results using a specific TQAPP. Liquid coating and environmental performance will be evaluated using EPA, ASTM, and other nationally or industry-accepted testing procedures. Any reported process performance parameters will have been generated and evaluated according to standard best engineering practices.

Additional assurance of comparability will be derived from the routine use of precision and accuracy indicators as described above, the use of standardized and accepted methods, and the traceability of reference materials.

4.3.2 Representativeness

The limiting factor to representativeness is the availability of a large sample population. Experimental designs will be constructed such that projects will either have sufficiently large sample populations per trial or statistically significant fractional populations. The tests will be conducted at the paint and equipment supplier-recommended operating conditions. If the test data obtained from standard materials meet the quantitative QA criteria (precision, accuracy, and completeness), the measurements of the tested samples will be considered representative of the coating technologies under evaluation and used to interpret the outcomes relative to the specific project objectives.

4.4 Other QA Objectives

No other QA objectives have been identified as part of this evaluation.

4.5 Impact of Quality

Due to the highly controllable nature of the test panel evaluation methods and predictability of factors affecting the quality of the laboratory testing of panels, the quality control of test panel qualifications is expected to fall within acceptable levels. Comparison of response factors will be checked for run-to-run process variations.

5.0 SITE SELECTION AND SAMPLING PROCEDURES

5.1 Site Selection

The exact test site location will depend on the equipment needed to complete the verification testing. Where possible, verification testing and the associated analyses will be conducted at *CTC*. At *CTC*, coatings will be applied and evaluated in the Demonstration Factory at the Environmental Technology Facility (ETF). All verification testing will be performed under the direct control of the Engineering and Statistical Support and Organic Finishing Line Groups. All laboratory analyses, where possible, will be performed in the *CTC* Testing Laboratory at the ETF.

In the event that *CTC* does not possess the application equipment recommended by the coating vendor, and the ETV CCEP does not choose to obtain it, *CTC* will choose a suitable offsite facility to run the verification test. At a minimum, the offsite facility must meet the standards of the individual TQAPP, the ETV CCEP QMP, and the ETV Program QMP. Any offsite facility chosen by *CTC* must, at a minimum, be able to clean, coat, cure, and adequately package finished parts and panels for shipment without damage. If the offsite facility has the capability to pretreat the panels, it is desirable to have the offsite facility pretreat the parts/panels. *CTC* will be responsible for laboratory analyses of the critical response factors, and the offsite testing site will be responsible for all critical, noncritical, and qualitative control factor data.

In the event that testing is performed at an offsite facility, a technical representative from *CTC* will be present during the test runs to ensure the quality of the critical, noncritical, and qualitative control factor data.

5.2 Site Description

Figure 2, in Section 2.1.1, illustrates the overall layout of *CTC*'s Demonstration Factory and the location of the process equipment that may be used for this protocol. The testing in this project involves the use of the pretreatment process with an associated dryoff oven, the liquid spray booths, and the wet cure oven. Other equipment or testing sites may be used, as necessary.

5.3 Sampling Procedures and Handling

Test panels and/or vendor-supplied parts will be used in this project. These will be prelabeled with a unique alphanumeric identifier stamp. The number of test panels processed during the testing depends on the experimental design. The experimental design, in turn, depends on the liquid coating vendor's claim(s) about performance characteristics and the respective confidence levels given in the responses to the RFT. If the coating vendor makes no performance claims, the default experimental design will be used. The default experimental design will consist of 40 samples (five runs with eight samples per run).

For testing conducted at *CTC*, the factory operations technician will process the test panels according to a preplanned sequence of stages, including pretreatment, application of the coating to the test panels, curing, and cooling, based on the coating vendor's recommended equipment settings. A laboratory analyst will record the date and time of processing and the process conditions. When the panels are removed from the racks, they will be separated by a layer of packing material and then stacked for transport to the laboratory. A laboratory analyst will take possession of the test panels from the factory operations technician and process the test panels through the laboratory login. Test panels taken to the laboratory for analysis will be labeled with the date, time, and initials of the laboratory personnel receiving the panels, and given a unique laboratory identification number.

For testing conducted offsite, *CTC* will ship either pretreated or untreated panels to the site. At the site, an operations technician (or equivalent) will process the panels according to a preplanned sequence of stages. A technical representative from *CTC* will be present to oversee these operations. The technical representative from *CTC* will be responsible for noting sampling time/date and initialing the sample log. After the panels are coated, cured, and cooled, they will be packaged and shipped back to *CTC*. A *CTC* laboratory analyst will receive, log (including the sampling time and date, as recorded by the *CTC* technical representative during testing), and analyze the finished panels.

When selecting a sampling site on the panels, consideration will be given to the following, as specified in the experimental design:

- Population size and reason for selection
- Description of sample type (whether panels, parts, wastes, etc.)
- Type of sampling strategy (whether simple, stratified, etc.)
- Statistical methods used and rationale
- Frequency and number of samples taken
- Location of sampling sites on the panels may be specified by the test method used
- Sources of contamination
- Effects of site selection on data validity.

5.4 Sample Custody, Storage, and Identification

All test panels will be given to the laboratory for login and assigned a unique laboratory ID number. The analyst delivering the test panels will complete a custody log indicating the sampling point IDs, sample material IDs, quantity of samples, time, date, and analyst's initials. The product evaluation tests will also be noted on the custody log. The laboratory's sample custodian will verify this information. Both personnel will sign the custody log to indicate transfer of the samples from the coating processing area at *CTC* or receiving area (for panels processed offsite) to the laboratory analysis area. The laboratory sample custodian will log the test panels into a bound record book; store the test panels under appropriate conditions (ambient room temperature and humidity); and create a work order for the various laboratory departments to initiate testing. The laboratory analyses will begin within several days of coating application.

6.0 ANALYTICAL PROCEDURES AND CALIBRATION

6.1 Facility and Laboratory Testing and Calibration

CTC has developed and currently maintains a calibration system within the factory and the laboratory. Testing and measuring equipment are calibrated on a periodic basis to ensure the accuracy of the data collected.

6.1.1 Facility Testing and Calibration

Calibration procedures within the factory are derived from ISO 10012-1 and MIL SPEC 45662A guidelines. A software package is used to track calibration information for each piece of testing and measuring equipment. This software serves to alert personnel when each piece of equipment is scheduled for calibration. Certified solutions and reference materials traceable to NIST are purchased when they are available. Where a suitable source of material does not exist, a secondary standard is prepared, and a true value is obtained by measurement against a NIST-traceable standard.

6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed at *CTC* are adapted from standard ASTM, MIL-SPEC, EPA, Association of Official Analytical Chemists (AOAC), and/or industry protocols for similar manufacturing operations. Initial calibration and periodic calibration verification are performed at the frequencies specified by the methodology to ensure that an instrument is operating sufficiently to meet sensitivity and selectivity requirements. At a minimum, all equipment is calibrated before use and is verified during use and/or immediately after each sample batch. Standard solutions are purchased from reputable chemical supply houses in diluted forms. Where certified and traceable-to-NIST reference materials and solutions are available, the laboratory purchases these for calibration and standardization. Data from all equipment calibrations and chemical standard certificates from vendors are stored in laboratory files and are readily retrievable. Each calibration procedure is documented as a formal standard laboratory operating procedure for which the analyst conducting experiments is trained. The analyst is also trained to detect nonconforming calibrations from method-specific QA checks. No samples are reported in which the full calibration curve, or the periodic calibration check standards, are outside method performance standards.

6.2 Product Quality Procedures

Each apparatus that will be used to assess the quality of a coating on a test panel is set up and maintained according to each manufacturer's, and/or the published reference method's, instructions. Actual sample analysis will take place only after set up is verified per the reference method and the equipment manufacturer's instructions. As available, samples of known materials with established product qualities are used to verify that a system is functioning properly. For example, traceable thickness standards are used to calibrate the eddy current thickness instrument. Applicable ASTM methods are listed in Appendix E.

6.3 Work Instructions (SOPs) and Calibration

Tables 10 and 11 summarize the methods and calibration criteria that will be used for the evaluation of the coatings. For every test performed routinely, the laboratory creates a standard operating procedure (SOP). SOPs are adapted from published references, such as ASTM and EPA, and from accepted protocols provided by industrial suppliers. SOPs are in the form of ISO 9000 Work Instructions. Work Instructions are created for equipment operation/sample analysis instructions, calibration, and maintenance. The *CTC* Laboratory Manager ensures that Work Instructions are created, reviewed, and followed by laboratory personnel. Each Work Instruction adheres to the quality elements contained in the original reference sources. The format for a laboratory Work Instruction is as follows:

- Title, Controlled ID #, Revision #
- Purpose
- Applicability
- Summary of Method
- Definitions
- Supporting Documents
- Equipment and Materials
- Training
- Environment, Health and Safety
- Calibration and Verification
- Maintenance
- Instruction/Process.

6.4 Nonstandard Methods

For nonstandard methods (i.e., no commonly accepted/specified method or traceable calibration materials exist), procedures will be performed according to the manufacturer's instructions or to the best capabilities of the equipment and the laboratory. This information will be documented in a SOP format. The performance will be judged based on the manufacturer's specifications or on inhouse-developed protocols. These protocols will be similar or representative in magnitude and scope to related methods performed in the laboratory that have reference performance criteria for precision and accuracy. For example, if a nonstandard quantitative chemical procedure is being performed, it should produce replicate results of +/- 25 relative percent difference and should give values within +/- 20 percent of true or expected values for calibration and percent recovery check samples. For qualitative procedures, replicate results should agree in terms of their final evaluations of quality or performance (i.e., both should either pass or both should fail if sampled together from a properly functioning process). The intended use and any limitations would be explained in a SOP for a nonstandard procedure. For this project, however, *CTC* does not intend to use any nonstandard methods.

Table 10. Noncritical Control Factor Testing and Calibration Criteria

Noncritical Factor	Method	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria[a]
Input Air Pressure	Factory Gauge	Pressure Gauge	Comparison to NIST-traceable standard	Six months	±5 psig
Pretreatment Weight	TBD	TBD	TBD	TBD	TBD
Surface Area of Each Panel	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	±0.1 cm
Ambient Factory Relative Humidity	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	±2% of full scale
Ambient Factory Temperature	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	±0.3 °C
Spray Booth Relative Humidity	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	±2% of full scale
Spray Booth Temperature	Thermal Hygrometer	Thermal Hygrometer	Sent for calibration or certification	Annually	±0.3 °C
Spray Booth Air Velocity	per ACGIH	Anemometer	Sent for calibration or certification	Annually	±5 above 0.5 m/s
Distance to Panels	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	±0.1 cm
Temperature of Panels, as Coated	IR Thermometer	IR Thermometer	Sent for calibration or certification	Annually	±0.5 °C
Horizontal Gun Traverse Speed	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	±0.001%
Vertical Drop Between Passes	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	±0.1 cm
Dwell Time Between Passes	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	±0.001%
Density of Applied Coating	ASTM D 1475	Weight	Comparison to NIST-traceable standard	With each use	±0.003 g
% Solids of Applied Coating	ASTM D 2369	Weight	Comparison to NIST-traceable standard	With each use	±0.003 g
Coating Temperature, as Applied	Thermometer	Thermometer	Comparison to NIST-traceable standard	Annually	±1 °C
Coating Viscosity, as Applied	ASTM D 1200	Ford Cup	Comparison to NIST-traceable standard	Prior to each test	±10%
Paint Flow Rate	Manufacturer's recommendation	Flow Meter	Comparison to NIST-traceable standard	Six months	±1% of full scale
Total Paint Flow	Manufacturer's recommendation	Flow Meter	Comparison to NIST-traceable standard	Six months	±1% of full scale
Oven Temperature	Thermocouple	Thermocouple/ (Controllers)	Comparison to NIST-traceable standard	Annually/ (Six months)	±2.2 °C/ (±0.8 °C)
Oven Cure Time	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	±0.001%

[a] As a percent recovery of a standard.
N/A - Not Applicable
TBD - To Be Determined

Table 11. Critical Response Factor Testing and Calibration Criteria

Critical Measurement	Method Number[a]	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria[b]
VOC Content	EPA Method 24	Volatile Content	Comparison to NIST-traceable standard	With each use	±0.003 g
HAP Content	EPA Method 311 or 8260B	GC/MS	Comparison to NIST-traceable standard	With each use	±0.003 g
DFT	ASTM B 499	Magnetic	Multipoint curve with NIST-traceable standards	Each use, verify calibration after 10 samples	90–110%
Visual Appearance	N/A	Visual	N/A	N/A	N/A
Gloss	ASTM D 523	Glossmeter	Multipoint curve with NIST-traceable standards	Each use, verify after each run	90–110%
Color	ASTM D 1729	Visual	N/A	N/A	N/A
Color	ASTM D 2244	Spectrometer	Zero with white tile	Each use	N/A
DOI[c]	ASTM D 5767 Method B[d]	Image analyzer	Manufacturer's recommendation	Manufacturer's recommendation	Manufacturer's recommendation
Adhesion	ASTM D 3359	Tape Test	Verify condition of scribes and freshness of adhesives	Each use	N/A
Pencil Hardness	ASTM D 3363	Pencil	Supplier graded lead (use same supplier)	Each use	N/A
Salt Spray	ASTM B 117	Salt Fog 5% NaCl Neutral pH	Verify collection rate, pH, salinity, and bare steel corrosion rate	Weekly chemical tests, monthly steel tests	rsd ≤20% among steel panels, avg. of chemical tests within specific ranges
Impact	ASTM D 2794 (Direct & Reverse)	2 lb weight	Verify weight of indentor, verify ruler	Yearly	80–120%
Flexibility	ASTM D 522 (Mandrel Bend)	Conical Mandrel	Verify conical diameter	Yearly	80–120%
MEK Rub	ASTM D 5402	MEK-Saturated Cheesecloth	Reagent grade MEK	N/A	N/A
Humidity Resistance	ASTM D 1735	100% Humidity using Fog App.	Collection rate, pH	Daily collection rate and pH	Must be within specified ranges
Weather Resistance	ASTM G 26	Xenon arc with and without humidity	Irradiance, temperature, black panel, wet and dry bulb, wattage, water quality	Weekly	Must be within specified ranges
Abrasion Resistance	ASTM D 4060	Taber Abraser	Verify load weights	Each use	95–105%

[a] Listing of ASTM methods to be used is provided in Appendix E.
[b] As a percent recovery of a standard.
[c] Performed by ACT Laboratories, Inc.
[d] Except: rotating eight-bladed disc replaces sliding combed shutter.
N/A - Not Applicable
TBD - To Be Determined

7.0 DATA REDUCTION, VALIDATION, AND REPORTING

7.1 Raw Data Handling

Raw data will be generated and collected by the analysts at the bench and/or process level. Process data is recorded into a process log during factory operations. Bench data will include original observations, printouts, and readouts from equipment for sample, standard, and reference QC analyses. Data will be collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst ID, raw or processed signal, and/or qualitative observations will be recorded. Comments documenting unusual or nonstandard observations will also be included on the forms, as necessary. Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being entered into a computer. The analyst will be responsible for scrutinizing the data according to specified precision, accuracy, and completeness policies. Raw data bench sheets, calculations, and data summary sheets will be maintained for each sample batch. From the written SOP and raw data bench files, the steps leading to a final result may be traced.

7.2 Preliminary Data Package Validation

The generating analyst will assemble a preliminary data package containing the QC and raw data results, calculations, electronic printouts, conclusions, and laboratory sample tracking information. A second analyst will review the entire package and may also check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to ensure that tracking, sample treatments, and calculations are correct. After the package has been peer reviewed in this manner, a preliminary data report will be prepared. The entire package and final report will be submitted to the *CTC* Laboratory Manager.

7.3 Final Data Validation

The *CTC* Laboratory Manager shall ultimately be responsible for all final data released from the laboratory. The *CTC* Laboratory Manager will review the final results for adherence to project QA objectives. If the *CTC* Laboratory Manager suspects an anomaly or nonconcurrence with expected or historical performance values, project QA objectives, or method-specific QA requirements of the laboratory SOP, he or she will initiate a second review of the raw data and query the generating and reviewing analysts about the nonconformance. The *CTC* Laboratory Manager will also request a specific corrective action. If suspicion about data validity still exists after internal review of laboratory records, the *CTC* Laboratory Manager may authorize a reanalysis. If a sufficient sample is not available for retesting, a resampling will occur. If the sampling window has passed or resampling is not possible, the *CTC* Laboratory Manager will flag the data as suspect and notify the *CTC* Project Manager. The *CTC* Laboratory Manager will sign and date the final data package.

7.4 Data Reduction

The liquid coatings being verified will be evaluated for VOC/HAP content during application and VOC/HAP emissions during curing, pending the approval of the new EPA method for determining cure emissions. In the event that the EPA method for determining the VOC/HAP emissions during the curing of the coating is approved, the analysis of those emissions may be included as a critical response factor. Liquid coatings will also be verified for finish quality and performance of the cured coating on standard test panels.

The results of these verification tests will be presented in the Verification Statement as environmental and performance factors. The environmental factors will include the VOC/HAP content determined during application and possibly VOC/HAP emissions given off during curing. The performance factors will include the results of the DFT, visual appearance and the selected optional performance analyses.

7.5 Data Reporting and Archival

The data generated by the liquid coating verification tests will be reviewed and validated by ETV CCEP project staff, the *CTC* QA Officer, and *CTC* Technical Peer Reviewers. This process constitutes data validation as required by the ETV CCEP QMP. After the data have been validated, a report signed and dated by the *CTC* Laboratory Manager is submitted to the *CTC* Project Manager, the *CTC* QA Officer, and other technical principals involved in the project. The *CTC* Project Manager will determine the appropriateness of the data and make any interpretations with respect to project QA objectives. The final laboratory report will contain the lab sample ID, date reported, date analyzed, the analyst, the SOP used for each parameter, the process or sampling point identification, the final result, and the units. The laboratory will retain the data packages for at least 10 years. The *CTC* Project Manager or the NDCEE Program Manager will forward the results and conclusions to the U.S. Army ARDEC and EPA in their regular reports, after obtaining corporate approvals.

The ETV CCEP will then prepare a Verification Report that includes a description of the tests performed, data obtained from those tests, and the calculations made from that data. The Verification Report will also be summarized as a Verification Statement. The raw data, results of data reduction, and QA analyses will be compiled into a separate Data Notebook.

The Verification Report, Verification Statement, and Data Notebook will undergo a brief preliminary review by the ETV EPA Pilot Manager and the ETV EPA Pilot QA Manager for format and consistency with the test's conclusions and for final data validation. They will then be reviewed by the liquid coating provider. The coating provider's comments will be incorporated into the test documents. The Verification Report, Verification Statement, and Data Notebook will then be distributed for *CTC* Technical Peer Review. Comments received from the *CTC* Technical Peer Reviewers will be addressed by *CTC* and the resolutions documented in writing. A revised Verification Report, Verification Statement, and a copy of the written documentation of the comment resolutions will then be submitted to the U.S. Army ARDEC and the ETV EPA Pilot Manager. The ETV EPA Pilot Manager will arrange for EPA Technical Editor review. The Data Notebook will not be reviewed by the EPA Technical Editor, but will instead be archived by *CTC* for future retrieval upon public or EPA request. Approval by EPA management will be coordinated by the ETV EPA Pilot Manager and the ETV EPA Program Manager. *CTC* will prepare the approved Verification Report and Verification Statement for posting on the ETV website. *CTC* will be responsible for publishing each Verification Report and Statement.

7.6 Verification Statement

After the EPA reviews the results and conclusions from the *CTC* Project Manager, the Verification Statement/Verification Report will be written by *CTC*, sent to the vendor for comment, passed through *CTC* Technical Peer Review, and submitted to the U.S. Army ARDEC, and presented to EPA for approval. Following agreement by the coating provider, *CTC* will disseminate the Verification Statement, which is a summary of the test results included in the Verification Report.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Guide Used for Internal Quality Program

CTC has established an ISO 9001 operating program for its laboratories and the Demonstration Factory. The laboratory is currently establishing a formal quality control program for its specific operations. The format for laboratory QA/QC is being adapted from several sources, as listed in Table 12.

Table 12. *CTC* Laboratory QA/QC Format Sources

Document	Reference Source
General Requirements for the Competence of Calibration and Testing Laboratories	ISO Guide 25, ISO Quality Programs
Critical Elements for Laboratories	Pennsylvania Department of Environmental Protection
Chapter One, Quality Control	SW-846, EPA Test Methods
Requirements of 100-300 series of methods	EPA Test Methods
Handbook of Quality Assurance for the Analytical Chemistry Laboratory, 2nd Ed.	James P. Dux

8.2 Types of QA Checks

The ETF laboratory at *CTC* follows published methodologies, where possible, for testing protocols. Laboratory methods are adapted from Federal Specifications, Military Specifications, ASTM Test Methods, and supplier instructions. The ETF laboratory adheres to the QA/QC requirements specified in these documents. In addition, where QA/QC criteria are not specified, or where the laboratory performs additional QA/QC activities, these protocols are explained in the laboratory's SOPs (Work Instructions). Each *CTC* facility that uses supplied products implements its own level of QA/QC. *CTC*'s laboratory at ETF will perform the testing and QA/QC verification outlined in Tables 8 and 9 (Precision, Accuracy, Completeness) and Tables 10 and 11 (Calibration); therefore, these tables should be referred to for the method-specific QA/QC that will be performed.

8.3 Basic QA Checks

During each test, laboratory staff will complete an internal Process QA Checklist to ensure that the appropriate parts, panels, samples, and operating conditions are used. The laboratory also monitors its reagent deionized water to ensure it meets purity levels consistent with analytical methodologies. The filters are replaced quarterly before failures are encountered. When failures do occur, samples are not processed until the filters are replaced. The quality of the water is assessed with method reagent water blanks. Blank levels must not exceed minimum detection levels for a given parameter to be considered valid for use.

Thermometers are checked against NIST-certified thermometers at two temperatures. The laboratory checks and records the temperatures of sample storage areas, ovens, hot plate operations, and certain liquid baths using thermometers.

Balances are calibrated by an outside organization using standards traceable to NIST. *CTC* also performs inhouse, periodic verifications with ASTM Class 1 weights. The ETF laboratory maintains records of the verification activities and calibration certificates. The laboratory analyst also checks the balances prior to use with ASTM Class 1 weights.

Reagents purchased directly by the laboratory are American Chemical Society (ACS) grade or better. Reagents, dated on receipt and when first opened, are not used beyond their certified expiration dates.

Laboratory waste is segregated according to chemical classifications in labeled containers to avoid cross-contamination of samples.

8.4 Specific Checks

CTC's ETF laboratory personnel will perform duplicate analyses on the same samples and calibration checks of the laboratory equipment. Laboratory personnel will also check any referenced materials and equipment as available and specified by the referenced methodology and/or the project-specific QA/QC objectives. Laboratory records are maintained with the sample data packages and/or in centralized files, as appropriate. To ensure comparability, laboratory personnel will carefully control process conditions and perform product evaluation tests consistently for each specimen. The specific QA checks listed in Tables 8 through 11 provide the necessary data to determine whether process control and product testing objectives are being met. ASTM, Federal, and Military methods that are accepted in industry for product evaluations and supplier-endorsed methods for process control will be used for all critical measurements, thus satisfying the QA objective. A listing of the published methods that will be used for this protocol is included in Appendix D.

9.0 PERFORMANCE AND SYSTEM AUDITS

CTC has developed a system of internal and external audits to monitor both program and project performance. These include monthly managers' meetings and reports, financial statements, EPA reviews and stakeholders' meetings, and In-Process Reviews. The ETF laboratory also analyzes performance evaluation samples to maintain Pennsylvania Department of Environmental Protection Certification.

ISO Internal Audits

CTC has established its quality system based on ISO 9000 and 14000 and has implemented a system of ISO internal audits. This information will be used for internal purposes.

On-Site Visits

The ETV EPA Pilot Manager may visit *CTC* for an onsite visit during the execution of this project. All project, process, quality assurance, and laboratory testing information will be available for review.

EPA Audits

The EPA will periodically audit *CTC* during this project. All project, process, quality assurance, and laboratory testing information will be made available per the EPA's auditing procedures.

Technical Systems Audits

A listing of all coating equipment, laboratory measuring and testing devices and procedures, coating procedures, and a copy of the approved ETV QMP and approved ETV CCEP QMP will be given to the *CTC* QA Officer. The *CTC* QA Officer will conduct audits of demonstration and testing activities according to the ETV CCEP QMP. The results of this activity will be forwarded to EPA in reports from the NDCEE Program Manager or *CTC* Project Manager.

Audits of Data Quality

Peer review in the laboratory constitutes a process whereby two analysts review raw data generated at the bench level. After data are reduced, they undergo review by laboratory management. For this protocol, laboratory management will spot check 10 percent of the project data by performing a total review from raw to final results. This activity is performed in addition to the routine management review of all data. Records will be retained to indicate which data have been reviewed in this manner.

10.0 CALCULATION OF DATA QUALITY INDICATORS

10.1 Precision

Duplicates will be performed on separate samples, as well as on the same sample source, depending on the method being employed. In addition, the final result for a given test may be the arithmetic mean of several determinations on the part or matrix. In this case, duplicate precision calculations will be performed on the means. The following calculations will be used to assess the precision between duplicate measurements.

Relative Percent Difference (RPD) = [(C1 - C2) x 100%] / [(C1 + C2) / 2]

where: C1 = larger of the two observations
C2 = smaller of the two observations

Relative Standard Deviation (RSD) = (s/y) x 100%

where: s = standard deviation
y = mean of replicates.

10.2 Accuracy

Accuracy will be determined as percent recovery of a check standard, check sample, or matrix spike.

For matrix spikes and synthetic check samples:

Percent Recovery (%R) = 100% x [(S - U)/T]

where: S = observed concentration in spiked sample
U = observed concentration in unspiked sample
T = true value of spike added to sample.

For standard reference materials (srm) used as calibration checks:

% R = 100% x (C_m / C_{srm})

where: C_m = observed concentration of reference material
C_{srm} = theoretical value of srm.

10.3 Completeness

Percent Completeness (%C) = 100% x (V/T)

where: V = number of determinations judged valid
T = total number of determinations for a given method type.

10.4 Project Specific Indicators

Process control limit: range specified by supplier for a given process parameter.

11.0 CORRECTIVE ACTION

11.1 Routine Corrective Action

Routine corrective action will be undertaken in the event that a parameter in Tables 8 through 11 is outside the limits prescribed in these tables, or when a process parameter is beyond specified control limits. Some examples of nonconformances include invalid calibration data, inadvertent failure to perform method-specific QA tests, process control data outside specified control limits, and failed precision and/or accuracy indicators. Such nonconformances will be documented on a standard laboratory form. Corrective action involves taking any necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard form. Some nonconformances will be detected during analysis or sample processing, and can be rectified in real time at the bench level. Others may only be detected following completion of processing trial and/or sample analyses. Typically, these types of nonconformances are detected at the *CTC* Laboratory Manager level of data review. In all cases of nonconformance, the *CTC* Laboratory Manager will consider repeating the sample analysis as one method of corrective action. If an insufficient sample is available or the holding time has been exceeded, complete reprocessing may be ordered to generate new samples. Reprocessing will only be performed if the *CTC* Project Manager determines that the nonconformance will jeopardize the integrity of the conclusions to be drawn from the data. In all cases, a nonconformance will be rectified before sample processing and analysis continue. If corrective action does not restore the production or analytical system, causing a deviation from the ETV CCEP QMP, *CTC* will contact the ETV EPA Pilot Manager. In cases of routine nonconformance, EPA will be notified in the NDCEE Program Manager's or the *CTC* Project Manager's regular report to the ETV EPA Pilot Manager. A complete discussion will accompany each nonconformance.

11.2 Nonroutine Corrective Action

While not anticipated, activities such as internal audits by the *CTC* QA Officer and onsite visits by the ETV EPA Pilot Manager, may result in findings that contradict deliverables in the ETV CCEP QMP. In the event that nonconformances are detected by bodies outside the laboratory organizational unit, as for routine nonconformances, these problems will be rectified and documented prior to processing or analyzing further samples or specimens.

12.0 QUALITY CONTROL REPORTS TO MANAGEMENT

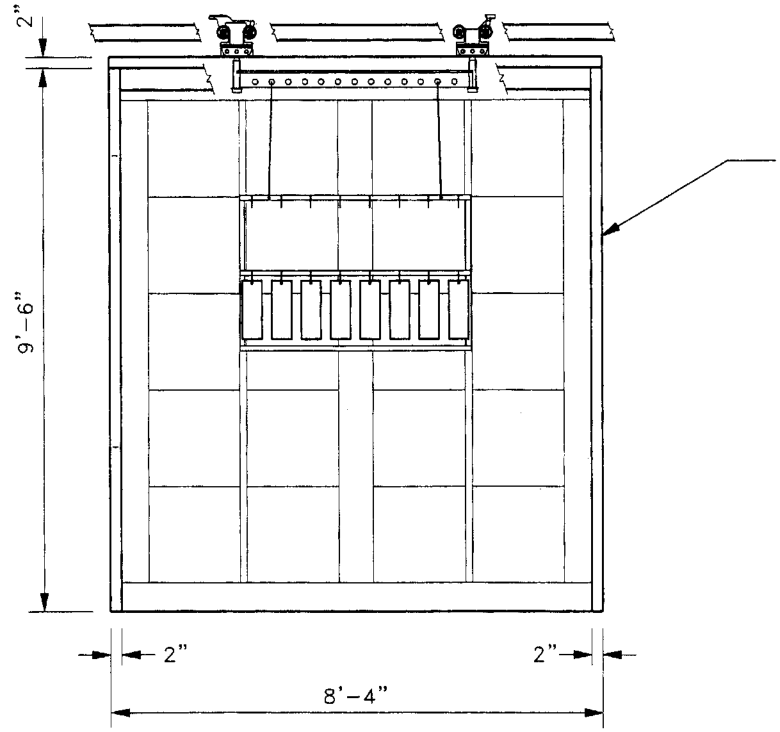
As shown on the Project Organization Chart in Figure 5, *CTC* employs a full-time QA Officer who is independent from the project management team. It is the responsibility of the *CTC* QA Officer to monitor *CTC* demonstration projects for adherence to project-specific QMPs. The *CTC* Laboratory Manager monitors the operation of the laboratory on a daily basis and provides comments to the *CTC* QA Officer to facilitate his or her activities. The *CTC* QA Officer will audit the operation records, laboratory records, and laboratory data reports, as well as provide a written report of his or her findings to the *CTC* Project Manager and *CTC* Laboratory Manager. The *CTC* Project Manager will ensure that these reports are included in the report to EPA. The *CTC* Laboratory Manager will be responsible for achieving closure on items addressed in the report. Specific items to be addressed and discussed in the QA report include the following:

- General assessment of data quality in terms of the general QA objectives in Section 4.1
- Specific assessment of data quality in terms of the quantitative and qualitative indicators listed in Sections 4.2 and 4.3
- Listing and summary of all nonconformances and/or deviations from the ETV CCEP QMP
- Impact of nonconformances on data quality
- Listing and summary of corrective actions
- Results of internal QA audits
- Closure of open items from the last report or communications with EPA in current reporting period
- Deviations or changes in the ETV CCEP QMP
- Progress of *CTC* QA Programs in relation to current project
- Limitations on conclusions, use of the data
- Planned QA activities, open items for next reporting period.

APPENDIX A

Apparatus Setup

ELEVATION VIEW – LIQUID SPRAY BOOTH WITH TEST PRODUCT



CTC Concurrent Technologies Corporation

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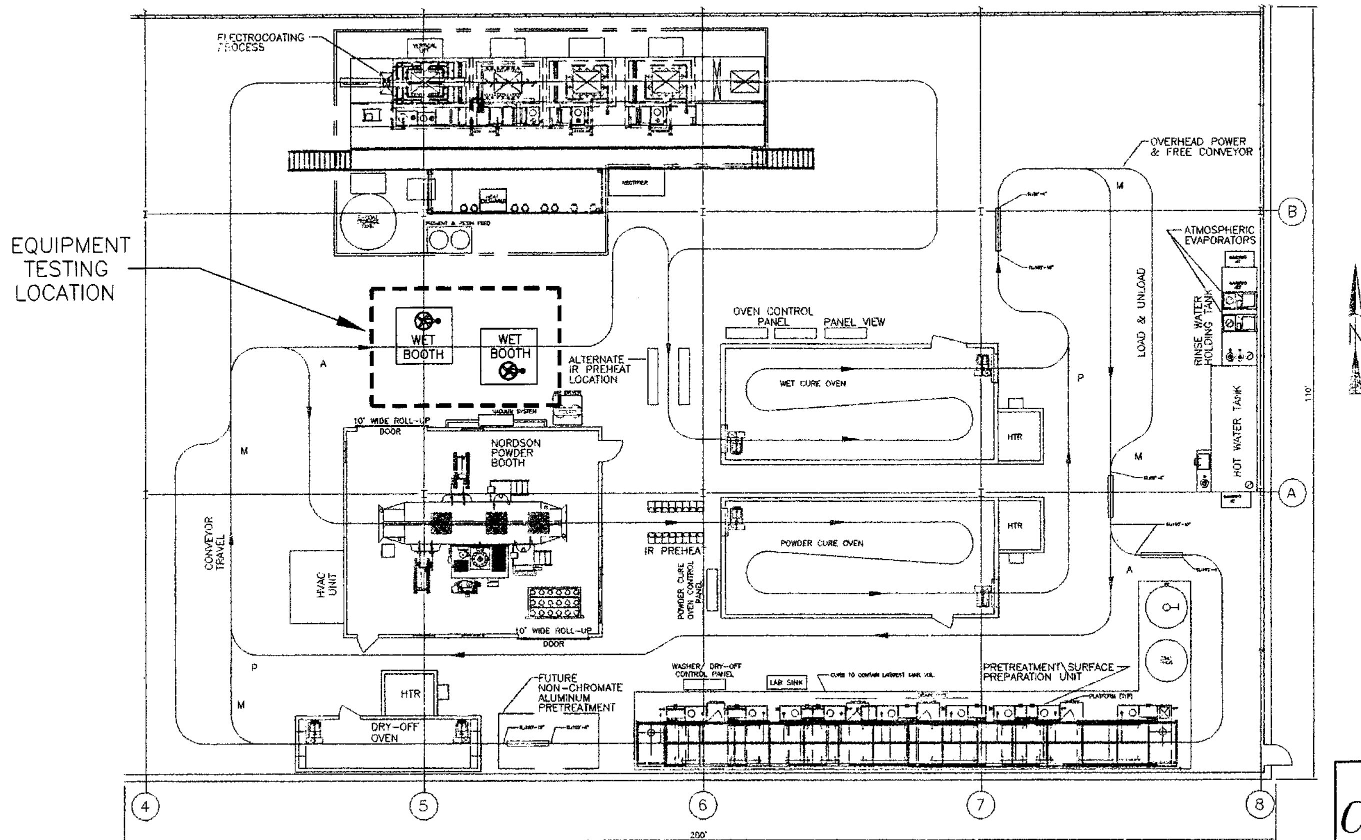
REVISIONS				
REV.	DATE	DESCRIPTION	BY	APPD

SCALE: NTS	PROJECT: ENVIRONMENTAL TECHNOLOGY VERIFICATION COATINGS AND COATING EQUIPMENT PROGRAM
ISSUE DATE: 12-12-97	
DRN BY: HHH	TITLE: TEST SET-UP DETAILS
CKD BY: BD	DRAWING NO.: ETF-060-A08
CKD BY:	REV. 0
APPD BY: MAH	

APPENDIX B

Equipment Testing Location



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REVISIONS				
REV.	DATE	DESCRIPTION	BY	APPD

SCALE: NTS
ISSUE DATE: 7-17-97
DRN BY: HHH
CKD BY: CMJ
CKD BY:
APPD BY: MSF

PROJECT: ENVIRONMENTAL TECHNOLOGY VERIFICATION COATINGS AND COATING EQUIPMENT PROGRAM

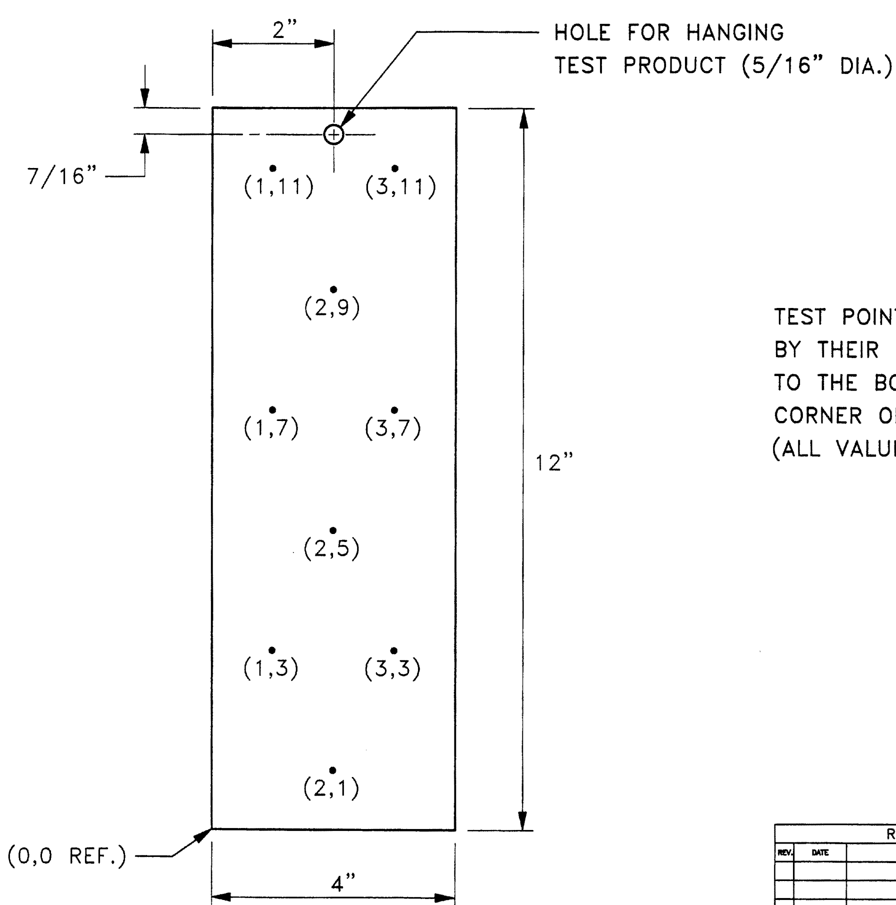
TITLE: LIQUID SPRAY EQUIPMENT TESTING LOCATION

DRAWING NO.: ETF-060-A03 REV. 0

APPENDIX C

Standard Test Panel

STANDARD TEST PRODUCT



TEST POINTS ARE INDICATED BY THEIR POSITION RELATIVE TO THE BOTTOM LEFT HAND CORNER OF THE PANEL. (ALL VALUES ARE IN INCHES).

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REVISIONS				
REV.	DATE	DESCRIPTION	BY	APPD

SCALE:	NTS
ISSUE DATE:	12-12-97
DRN BY:	HHH
CKD BY:	
CKD BY:	
APPD BY:	

PROJECT: ENVIRONMENTAL TECHNOLOGY VERIFICATION COATINGS AND COATING EQUIPMENT PROGRAM

TITLE: TEST PRODUCT WITH MEASUREMENT LOCATIONS

DRAWING NO.: ETF-060-A07

REV. 0

APPENDIX D

ASTM Methods

ASTM Methods

ASTM B 117	--	Standard Test Method of Salt Spray (Fog) Testing
ASTM B 499	--	Standard Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals
ASTM B 767	--	Standard Guide for Determining Mass per Unit Area of Electrodeposited and Related Coatings by Gravimetric and other Chemical Analysis Procedures
ASTM D 522	--	Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings
ASTM D 523	--	Standard Test Method for Specular Gloss
ASTM D 1200	--	Standard Test Method for Viscosity by Ford Viscosity Cup
ASTM D 1475	--	Standard Test Method for Density of Paint, Varnish, Lacquer, and Related Products
ASTM D 1729	--	Standard Practice for Visual Evaluation of Color Differences of Opaque Materials
ASTM D 1735	--	Standard Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus
ASTM D 2244	--	Standard Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates
ASTM D 2369	--	Standard Test Method for Volatile Content of Coatings
ASTM D 2794	--	Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)
ASTM D 3359	--	Standard Test Method of Salt Spray (Fog) Testing
ASTM D 3363	--	Standard Test Method for Film Hardness by Pencil Test
ASTM D 3960	--	Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings
ASTM D 4017	--	Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method
ASTM D 4060	--	Standard Test Methods for Abrasion Resistance of Organic Coatings by the Taber Abraser
ASTM D 4457	--	Standard Test Method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph
ASTM D 5402	--	Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs
ASTM D 5767	--	Standard Test Methods for Instrumental Measurement of Distinctness-of-Image Gloss of Coating Surfaces
ASTM E 1064	--	Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration
ASTM G 26	--	Practice for Operating Light Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials
EPA Method 24	--	Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings
EPA Method 311	--	Analysis of Hazardous Air Pollutant Compounds in Paints and Coatings by Direct Injection into a Gas Chromatograph
EPA Method 8260B	--	Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)